

**SPECIATION OF MANGANESE IN BLOOD SERUM -
ANALYTICAL METHODS DEVELOPMENT AND
OPTIMIZATION FOR THE EXTRACTION,
PRECONCENTRATION, AND DETERMINATION**

**A dissertation submitted to the Faculty of Science, University of the Witwatersrand,
Johannesburg, in fulfillment of the requirements for the degree of Master of
Science.**

By

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DECLARATION

I declare that this Dissertation is my own work, unaided work. It is being submitted for the Degree of Master of Science in the University of the Witwatersrand, Johannesburg. It has not been submitted before for any degree or examination in any other University.

Stephen Pole

_____ day of _____, 2005

Abstract

The analytical methods for speciation of manganese in three different types of matrices (water, milk and blood serum) was studied. Supported liquid membrane (SLM) extraction was optimized and successfully used for the extraction and preconcentration on Mn(II) from water, milk and blood serum. The extractant used was 15% (v/v) DEHPA with an organic membrane modifier, 10% (v/v) TOPO. All determinations of Mn(II) were carried out using Adsorptive Stripping Voltammetry.

A SLM membrane probe was developed and used for the extraction of Mn(II) from smaller water, milk and blood serum samples. Membrane probe depth was optimized: probe depth - 2mm below donor solution. The membrane probe yielded higher extraction efficiencies compared to the flat spiral disk SLM unit.

The Adsorptive Stripping Voltammetry method used for the determination of Mn(II) in water was optimized and used in other applications (determination of Mn(II) in aged and fractionated blood serum). The optimum conditions obtained: pH = 7.5-8.0, deposition potential = -(1.9V-2.0V), deposition time = 45-65s, equilibration time = 5s, stirring speed = 2000rpm, and no gelatin addition.

The AdSV optimized parameters were then used for the analysis of Mn(II) in aged whole and fractionated blood serum. Size exclusion chromatography was used to obtain the blood serum fractions. It was determined that with aging, the concentration and thus extraction efficiency of free Mn(II) in blood serum matrix decreases. The spiked blood serum fractions were extracted and Mn(II) was determined with adequate reproducibility.

DEDICATION

To my Mom, Dad, Goko,
And close friends.

You are all special!

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God for giving me the opportunity and strength to perform this research.

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LIST OF SYMBOLS

SLM Supported Liquid Membrane

LLE Liquid-Liquid Extraction

BLM Bulk Liquid Membrane

ELM Emulsion Liquid Membrane

PTFE Polytetrafluoroethylene

TOPO Tri-n-octylphosphine oxide

DEHPA Di-2-ethylhexyl phosphoric acid

SEC Size Exclusion Chromatography

GPC Gel Permeation Chromatography

SEM Scanning Electron Microscopy

AdSV Adsorptive Stripping Voltammetry

ASV Anodic Stripping Voltammetry

CSV Cathodic Stripping Voltammetry

ICP-OES Inductively Coupled Plasma Optical Emission Spectroscopy

ICP-AES Inductively Coupled Plasma Atomic Emission Spectroscopy

GF-AAS Graphite Furnace Atomic Absorption Spectroscopy

SOD Superoxide Dismutase

MnSOD Manganese Superoxide Dismutase

WHO World Health Organisation

BBB Blood brain barrier

Tf Transmanganin transferring

CNS Central Nervous System

Mp Melting point

E Extraction efficiency

E_e Enrichment factor

RSD Relative Standard Deviation

PGMs Platinum Group Metals

NCOH National Center for Occupational Health

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